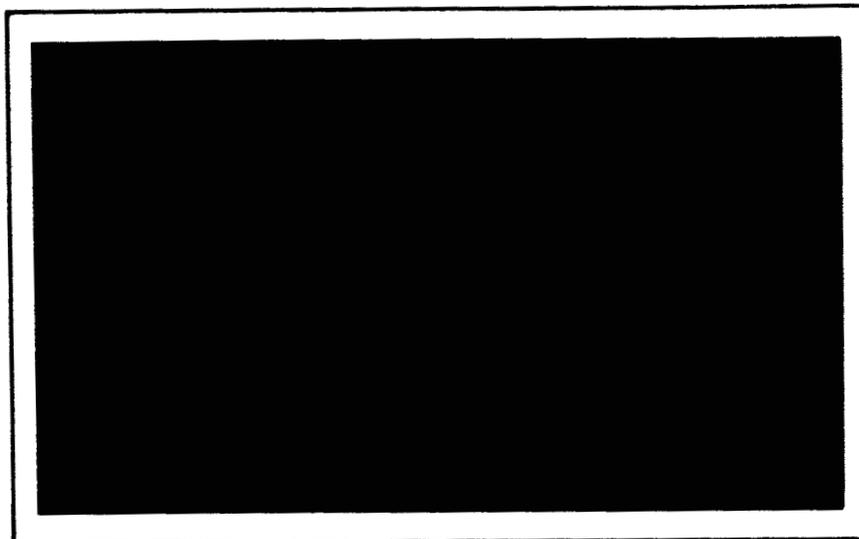


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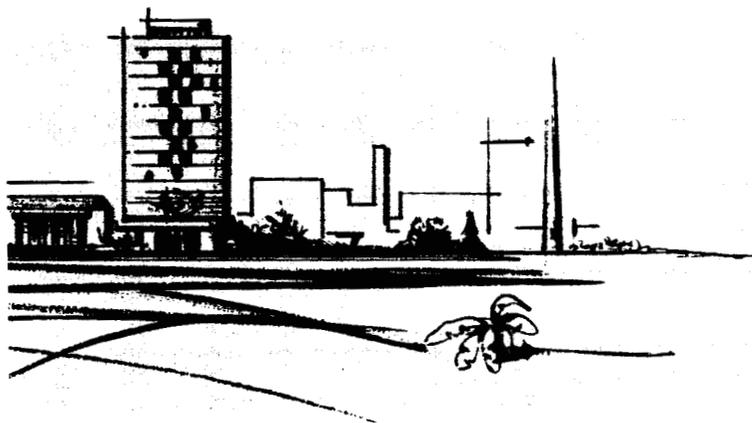


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ANNUAL SUMMARY REPORT

on

A STUDY OF HYDROGEN EMBRITTLEMENT
OF VARIOUS ALLOYS

to

NATIONAL AERONAUTICS AND
SPACE ADMINISTRATION
GEORGE C. MARSHALL
SPACE FLIGHT CENTER

June 23, 1966

by

T. P. Groeneveld, E. E. Fletcher, and A. R. Elsea

Ferrous and High Alloy Metallurgy Division
Process and Physical Metallurgy Department

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A STUDY OF HYDROGEN EMBRITTLEMENT OF VARIOUS ALLOYS

by

T. P. Groeneveld, E. E. Fletcher, and A. R. Elsea

ABSTRACT

The objectives of this program were (1) to determine the susceptibility of various specified alloys to hydrogen embrittlement, with particular attention given to embrittlement caused by cleaning, pickling, and electroplating processes, and (2) to determine the effectiveness of commonly used hydrogen-embrittlement relief treatments.

Hydrogen-stress-cracking experiments, hydrogen-entry-rate experiments, and hydrogen analyses were performed on specimens of the 14 selected materials cathodically charged under standard conditions. Five of the alloys evaluated (Ti-6Al-4V, Alloy 718, Waspaloy, René 41, and U-212 steel) were found to be insensitive to hydrogen-stress cracking under the conditions used; all the other materials were susceptible under the most severe hydrogen-charging condition. As the severity of the charging condition was reduced, differences in the relative susceptibilities to hydrogen-stress cracking among the various steels became more apparent. Comparison of the hydrogen-stress-cracking data and the results of the hydrogen-entry-rate experiments revealed that there was no simple direct correlation between hydrogen-entry rate and susceptibility to hydrogen-stress cracking. However, for six of the eight susceptible alloys for which the evaluation was completed, the susceptibility to hydrogen-stress cracking increased with increasing hydrogen-entry rate. The AISI 4340 and 4130 steels appeared to have abnormally low entry rates for the high susceptibility to cracking that they exhibited. The hydrogen analyses performed on specimens cathodically charged under the limiting conditions for occurrence of hydrogen-stress cracking showed that the various materials do have different hydrogen tolerance levels, i. e., they differ in the amount of hydrogen required to initiate failure. The results of these analyses also suggested that hydrogen distribution within the sample is more important than is average hydrogen content in determining susceptibility to failure.

Other experiments evaluated the hydrogen-stress-cracking behavior of notched tensile specimens of AISI Type H-11, AISI 4130, AISI 4340, AISI E 8740, and 18Ni maraging steels that had been cleaned and electroplated in cadmium cyanide baths with and without brighteners. The results showed that the preplating cleaning processes introduced sufficient hydrogen to cause embrittlement in some of the alloys. Also, both plating processes caused embrittlement in some of the alloys, although the dull-cadmium bath was less embrittling than was the bright-cadmium bath. Of the alloys evaluated, AISI 4340 steel was the most embrittled, and the 18Ni maraging steel was the least embrittled. Hydrogen analyses performed on specimens after the cleaning and electroplating processes revealed that, in general, the plating process itself introduced more hydrogen than did the cleaning process.

Specimens of 17-7 PH stainless steel, 17-4 PH stainless steel, and AM-355 stainless steel were evaluated after cleaning and electroplating in a Wood's-nickel-strike bath. The results of the hydrogen-stress-cracking experiments revealed that only the

17-7 PH stainless steel was embrittled by this processing. Hydrogen analyses again showed that the plating process introduced more hydrogen than did the cleaning process.

INTRODUCTION

When atomic hydrogen enters steel and certain other metals and alloys, it may cause any of several undesirable things to happen. If large quantities of hydrogen are introduced, there may be a general loss in ductility or, if the hydrogen accumulates in certain localized areas, internal bursts or blisters may be produced. At elevated temperatures, hydrogen may remove so much carbon from steel that the material is permanently weakened and is no longer capable of supporting the design stresses. Under certain circumstances, hydrogen may react with the alloy to produce catastrophic brittle failures at applied stresses far below the yield strength or the nominal design stress for the alloy. All of these phenomena are collectively referred to as hydrogen embrittlement. However, only the last of those mentioned, i. e., the hydrogen-induced catastrophic brittle failure of high-strength structural materials at relatively low applied stresses, is of interest in the present research program. Since this phenomenon frequently occurs in materials that exhibit no appreciable loss in ductility (as measured by a conventional tensile test), it is often termed hydrogen-induced delayed brittle failure or hydrogen-stress cracking. The latter term will be used in this discussion.

Hydrogen-stress cracking has been observed only in certain transition metals, including a number of metals with body-centered cubic structures and at least one hexagonal close-packed metal. Among the materials of interest that have been shown to be susceptible to hydrogen-stress cracking at ambient temperatures are ferritic and martensitic steels and certain titanium alloys.

It is generally agreed by the various investigators in this field that the hydrogen-stress cracking of steel is influenced by several factors. As the strength level of the steel is increased above some minimum value, as the applied stress is increased above some minimum (but much lower) value, and as the hydrogen content is increased, the time required for hydrogen-stress cracking to occur decreases. Also, there are indications that under given hydrogen-charging conditions, the maximum stress that the steel can support without failure decreases as the strength level of the steel is increased. In addition, it is generally agreed that hydrogen must be free to move through the steel before hydrogen-stress cracking will occur. This phenomenon is temperature dependent and appears to be most severe in the general vicinity of room temperature. At cryogenic temperatures where the diffusion rate of hydrogen is nil, hydrogen-stress cracking does not occur. At elevated temperatures, the diffusion rate of hydrogen is so rapid that a material may lose the damaging hydrogen before the mechanism can become operative. Hydrogen-stress cracking of steels is also strain-rate dependent, but not in the same way that most other forms of embrittlement are. Ordinarily, hydrogen has no effect on behavior of steel in an impact test. Some hydrogen-charged specimens exhibit normal ductility in a conventional tensile test, but at very low strain rates or under a static load the specimens may show very severe embrittlement. Thus the phenomenon of hydrogen-stress cracking is referred to as one of low-strain-rate embrittlement.

Relatively little work has been done on the austenitic grades of stainless steel or on the nickel-base alloys with face-centered cubic structures. Although these materials may show some loss in ductility after hydrogen charging, they usually are considered to be relatively insensitive to hydrogen-stress cracking. However, Troiano et al. (1)* recently showed that a nickel-base alloy, K-Monel, was sensitive to hydrogen-stress cracking at slightly elevated temperatures where the diffusion rate of hydrogen in the face-centered cubic lattice was comparable to the diffusion rate of hydrogen in the body-centered cubic lattice at room temperature. Cavett and Van Ness (2) found a 55 percent loss in notch tensile strength of precipitation-hardened K-Monel as the result of exposure to high-pressure hydrogen gas. Although no known instances of hydrogen-stress cracking of austenitic stainless steel in the bulk form are known to the authors of the report, Whiteman and Troiano (3) achieved failure in thin films of this material by charging under severe conditions. In spite of its high solubility for hydrogen, it appears to be extremely difficult to charge austenitic stainless steel with hydrogen beyond a very thin surface layer. Also, Wilcox and Smith (4) described the intercrystalline brittle fracture of hydrogen-charged nickel. Consequently, under severe environmental conditions, hydrogen-stress cracking may be a problem in these materials as well.

Titanium alloys exhibit two types of hydrogen embrittlement; these have been designated impact embrittlement and low-strain-rate embrittlement. The type of hydrogen embrittlement that is most often encountered in high-strength, alpha-beta titanium alloys is the low-strain-rate type. Sensitivity of titanium alloys to hydrogen embrittlement at low strain rates appears to increase with increasing tensile strength, notch severity, alpha grain size, continuity of the beta phase, and hydrogen content. Thus, in some respects, the hydrogen embrittlement of alpha-beta titanium alloys resembles that of high-strength steels.

In view of the preceding discussion, it would be expected that any condition that alters the strength, the applied stress, or the hydrogen content of a given material could influence the sensitivity to hydrogen embrittlement. The general trend toward high tensile strengths, higher design stresses, and the use of materials in applications requiring prolonged exposure to sustained loads insures that two of the conditions necessary for the occurrence of hydrogen-stress cracking will be present in certain types of aerospace and aircraft applications. All that remains is for an adequate supply of hydrogen to be available and for this hydrogen to be free to diffuse through the material. If the material is sensitive to hydrogen-stress cracking, delayed failures can be expected to occur. Thus, it appears that for high-strength materials sensitive to hydrogen-stress cracking, the most important factors tending to promote hydrogen-stress cracking under these conditions are the hydrogen content of the material and the propensity of the material to absorb hydrogen from its environment, either during processing or in service.

The source of the hydrogen in the metal is of little importance. It can be introduced during melting or heat-treating operations; during cleaning, pickling, or electroplating processes; or it may be picked up from the service environment as a result of cathodic protection reactions or corrosion reactions, for example. In this program, attention has been directed primarily to the hydrogen introduced into the materials during conventional cleaning, pickling, or electroplating processes.

A number of years ago, when several failures of electroplated high-strength-steel aircraft components were attributed to hydrogen embrittlement, the U. S. Air Force

*References are listed at the end of the report.

placed restrictions on the use of surface treatments on ultrahigh-strength steels if the treatments were suspected of introducing hydrogen into the steel. This action caused the manufacturers to resort to surface treatments that were inferior to electroplates from the standpoint of corrosion protection. As a result, considerable effort has been concentrated on the study of conventional cleaning and electroplating processes and the development of low-embrittling or nonembrittling cleaning and electroplating processes.

The results of various studies of conventional processing showed that the commonly used high-strength structural steels were susceptible to hydrogen-stress cracking resulting from hydrogen introduced during commonly employed cadmium-, chromium-, zinc-, nickel-, and tin-electroplating processes. Also, it was learned that frequently as much or more hydrogen was introduced into the steel during preliminary processing (such as cleaning, pickling, and activation processes) than was introduced by the electroplating operation itself.

If the hydrogen that has been introduced into steels during the various processing operations can be removed before permanent damage occurs, a part may be suitable for high-stress applications without fear of hydrogen-stress cracking, provided that additional hydrogen is not introduced by the service environment. It is common practice to subject electroplated high-strength steel parts to hydrogen-relief baking treatments. The diffusion rate of hydrogen in steel increases with increasing temperature; therefore, warming the steel tends to increase the rate of hydrogen removal. However, the solubility of hydrogen in steel also increases with increasing temperature, and experiments have shown that, for ferritic or martensitic steels, the optimum temperature for the removal of hydrogen is approximately 500 F. This temperature is in the range where other embrittling reactions can occur; in addition, if the steel part has been cadmium plated, damage to both the plate and the steel may result. Experience has shown that the most effective temperature for removing hydrogen from cadmium-plated high-strength steel parts is approximately 375 F, and it is usual practice to bake at that temperature for approximately 24 hours. However, a dense cadmium plate is an effective barrier to the removal of hydrogen by baking, and often such treatments are ineffective in eliminating the tendency for hydrogen-stress cracking to occur.

The most likely sources of hydrogen contamination in titanium alloys are chemical-milling operations and pickling processes in which the ratio of HF to HNO₃ is not properly controlled. Because of the high solubility and low diffusivity of hydrogen in titanium alloys, removal of hydrogen is normally not accomplished by baking operations familiar to users of high-strength steels. Although the hydrogen content of high-strength titanium alloys can be reduced by vacuum annealing at 1100 F or higher, the hydrogen content below which embrittlement does not occur has not been precisely determined. On the basis of standardized tests, the hydrogen tolerance has been approximated at 200 parts per million for alloys of the Ti-4Al-4Mn and Ti-6Al-4V types. This tolerance level is many times greater than the level required to cause hydrogen-stress cracking in high-strength steels.

OBJECTIVES

The objectives of this research program were (1) to investigate the susceptibility of selected high-strength structural alloys to hydrogen embrittlement, with particular

attention being given to hydrogen embrittlement induced by various pickling, cleaning, and electroplating processes and (2) to determine the effectiveness of commonly used hydrogen-embrittlement relief treatments.

The program consisted of two parts, (1) a review of the technical literature and current research activities in the area of hydrogen embrittlement and (2) a research program.

LITERATURE AND INDUSTRIAL SURVEY

Four documents issued by the Defense Metals Information Center⁽⁵⁻⁸⁾ provide a fairly comprehensive review of the information published on the general subject of hydrogen embrittlement through 1962. Since these documents were written by two of the personnel associated with this program, they served as the background for the survey.

To supplement these documents, various technical abstracts were searched for information pertaining to hydrogen embrittlement published since 1961. In addition, the DMIC files were searched for reports dealing with research activities in this area. The emphasis was placed on obtaining information dealing with hydrogen embrittlement of the alloys selected for investigation in the present research program; hydrogen embrittlement resulting from cleaning, electroplating, and other surface treatments; and hydrogen-embrittlement relief treatments.

The results of this survey, together with a summary of the DMIC documents, were used to prepare a literature review that was issued as a special report dated January 12, 1966⁽⁹⁾.

TECHNICAL APPROACH

The experimental program was divided into three phases. Phase 1 consisted of a preliminary study of the susceptibilities of the selected alloys to hydrogen-stress cracking. Those alloys found to be insensitive to hydrogen-stress cracking were dropped from the program, while those found to be susceptible were further evaluated in Phase 2 for susceptibility to hydrogen embrittlement resulting from various commonly used cleaning, pickling, and electroplating processes. In Phase 3, the effectiveness of various commonly used hydrogen-embrittlement relief treatments was to have been investigated. However, difficulties in obtaining and processing some of the materials delayed the experimental program, and work on Phase 3 was not initiated. This work will be taken up in the next term of this contract. The following paragraphs briefly describe the experiments performed during this contract period.

In Phase 1, the materials were evaluated for susceptibility to hydrogen-stress cracking by cathodically charging smooth tensile specimens of each material with hydrogen while they were under an applied stress of approximately 80 percent of their respective yield strengths. If a material sustained the applied stress for 200 hours

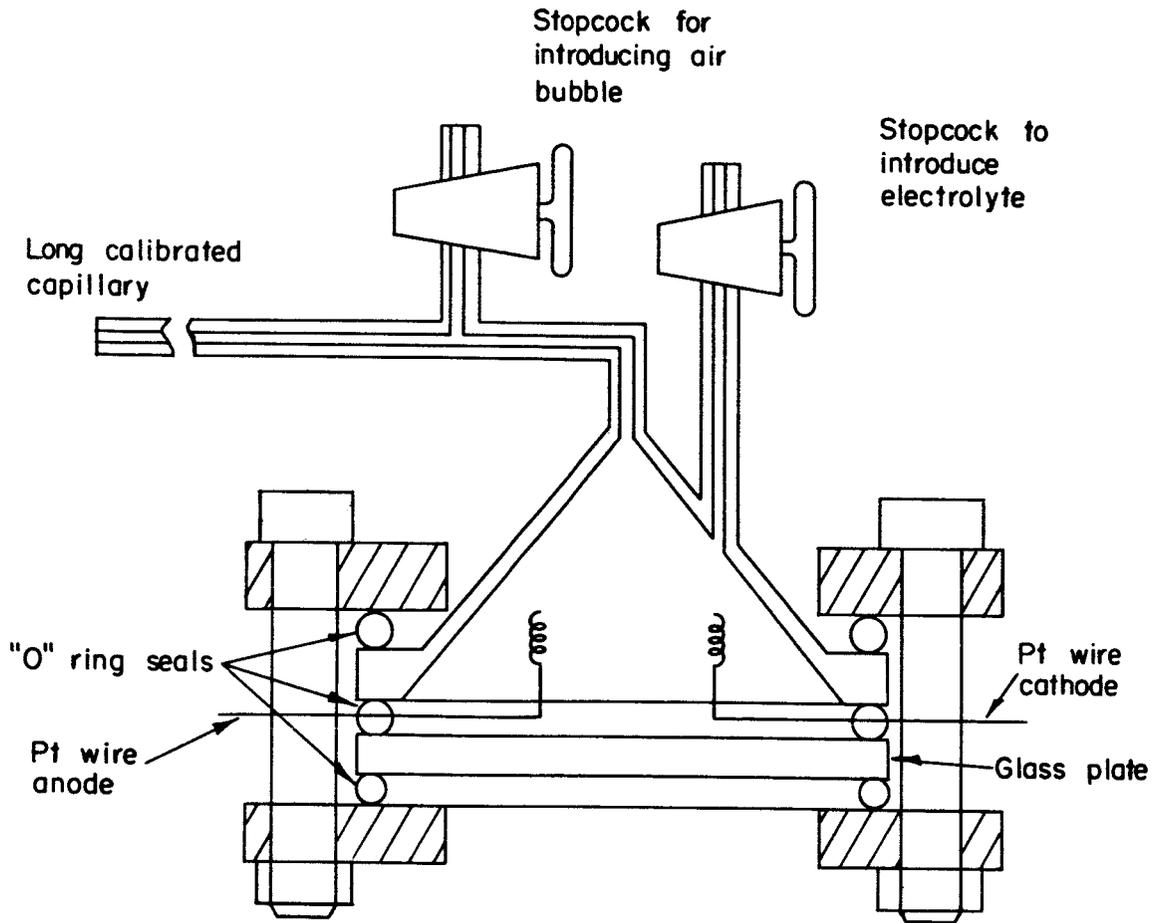
under the most severe charging condition used, it was considered insensitive to hydrogen-stress cracking at room temperature and was dropped from the program. The reasoning used to establish this criterion was that if the material did not fail under this charging condition, it is very unlikely that it would pick up enough hydrogen during conventional cleaning, pickling, or electroplating processes to cause hydrogen-stress cracking.

Those materials that failed under the most severe charging condition were then further evaluated under less severe conditions in an attempt to determine the limiting conditions under which hydrogen-stress cracking would occur, considering 200 hours to be the run-out time. Small samples of the material were then charged under the limiting conditions; one specimen was charged under the mildest conditions that produced a failure while another specimen was charged under the most severe conditions that did not produce a failure. Following charging, these samples were analyzed to determine the average hydrogen content.

Hydrogen-entry-rate experiments also were conducted to determine whether the rate at which a material accepts hydrogen is directly related to its susceptibility to hydrogen-stress cracking. These experiments were conducted using the apparatus shown in Figures 1 and 2. The apparatus consists of two cells of equal volume with a calibrated capillary tube attached to each cell. The cells and capillary tubes are filled with electrolyte and bubbles are introduced into the tubes for reference purposes. Displacement of the electrolyte, which results from gas evolution caused by electrolysis in the cells, can be measured by the displacement of the bubbles in the capillary tubes. Since the cells are connected electrically in series, the current flowing through them is identical and, therefore, with the same electrolyte the volume of gases produced by electrolysis should be the same in each cell. The current-efficiency cell, shown schematically in Figure 1, contains a platinum anode and a platinum cathode saturated with hydrogen; this cell measures the total volume of gas produced by electrolysis. The hydrogen-entry cell, shown schematically in Figure 2, contains a platinum anode, and the material under study serves as the cathode. Since the only difference in the cells is the material that constitutes the cathode, the difference between the displacement of the bubbles in the capillary tubes of the respective cells indicates the amount of hydrogen entering the specimen as a function of time.

The results of the Phase 1 experiments should provide a correlation between the susceptibility of the material to hydrogen-stress cracking, the rate at which the material accepts hydrogen, and the amount of hydrogen measured in the sample.

In Phase 2, notched tensile specimens of the alloys found to be susceptible to hydrogen-stress cracking in Phase 1 were evaluated for susceptibility to delayed failure as a result of hydrogen picked up during conventional cleaning, pickling, and electroplating procedures. The specimens were subjected to these types of treatments and then were subjected to a static tensile stress. The specimens that were subjected to the cleaning and activation processes were loaded to an applied stress equal to 90 percent of their respective notched tensile strengths. If the specimens did not fail in 100 hours, they were considered to be nonembrittled and were removed from the apparatus. The cleaned and electroplated specimens were initially loaded to 75 percent of their respective notched tensile strengths. If they survived 100 hours at this stress level, the stress was increased to 90 percent of the notched tensile strength. If they survived 100 hours at this stress level, they were considered to be nonembrittled and were removed from the apparatus.



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FIGURE 1. CURRENT EFFICIENCY CELL

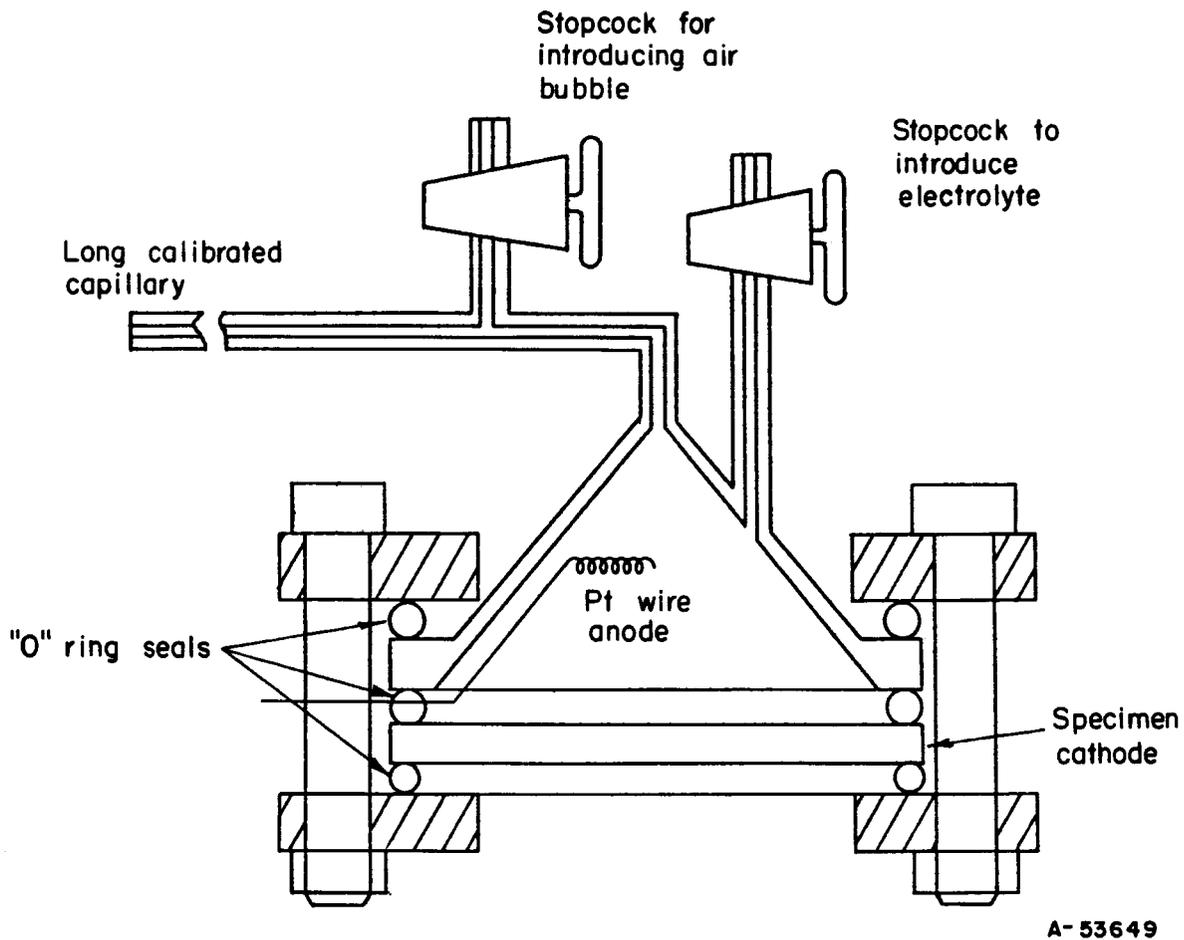


FIGURE 2. HYDROGEN ENTRY CELL

The reason for loading the cleaned specimens immediately to 90 percent of their notched tensile strength was that at room temperature hydrogen can readily diffuse out of steels. Therefore, unless the specimens were severely embrittled, by testing at a lower applied stress where it would take longer to produce delayed failures, they might lose sufficient hydrogen during the test time so that the failure mechanism could not become operative. This result would indicate (erroneously) that they were not embrittled. The 90 percent applied stress level represents a severe loading condition and would be expected to detect even moderate embrittlement.

In addition to the hydrogen-stress-cracking experiments employing cleaned and electroplated specimens, hydrogen analyses were conducted on samples of each material that were subjected to the same processes. Comparison of these hydrogen contents with those obtained from Phase 1 should indicate whether the selected process introduced more hydrogen than the critical amount determined to be required to produce failure under the loading conditions and runout time used in Phase 1. Also, these results should indicate whether the preplating cleaning and activation processes or the electroplating process itself was responsible for introducing the greater amount of hydrogen.

MATERIALS AND SAMPLE PREPARATION

Procurement

Fourteen alloys and the respective strength levels at which they were to be evaluated in this program were selected by NASA personnel. These alloys, hereafter referred to as the specified alloys, and the strength levels are listed in Table 1. In discussing the desired strength levels that were specified and the heat treatments that were indicated in a few cases, certain changes were mutually agreed upon so that the properties would be in line with those that can be readily achieved and so that the heat treatments would correspond to the properties to be expected.

A sufficient quantity of each of the specified alloys was obtained so that all samples of a given material to be used in this program could be machined from a single lot. To facilitate sample preparation and to comply with NASA requests, initial efforts were directed toward obtaining the materials in the form of 1/2-inch-thick plate or 1/2 x 3-inch bar stock. However, four of the alloys (AISI E 8740 steel, AM-355 stainless steel, Ti-6Al-4V, and U-212 steel) were not readily available in the desired form. Therefore, other forms of these materials were obtained, and they were processed in the laboratory to the desired 1/2-inch-thick bar as follows:

- (1) AISI E 8740 steel, received as 1-7/8-inch-diameter bar stock. The bar was cut into 16-inch lengths that were heated to 2150 F and forged into bars 1 inch thick and 3 inches wide. The forged bars were then reheated to 2150 F and hot rolled to a thickness of 0.50 inch, taking 10 percent reduction per pass and reheating after each pass. The finishing temperature was approximately 1900 F after final hot rolling, and the bars were air cooled to room temperature.

- (2) AM-355 stainless steel, received as 1-1/2 x 1-1/2 x 68-inch bar stock. The bar was cut into 10-inch lengths that were heated to 2150 F and pressed to a thickness of 1 inch in a 700-ton press. The pressing operation was performed in such a way as to produce the desired 3-inch width. The bars were then reheated to 2150 F and hot rolled to a thickness of 0.580 inch, taking 10 percent reduction per pass and reheating after each pass. A final pass was made at 1900 F to produce a finished thickness of 0.525 inch, following which the bars were air cooled to room temperature.
- (3) Ti-6Al-4V, received as 2 x 4 x 12-inch and 1-1/2 x 3-1/2 x 15-inch bars. The bars were heated to 1800 F and rolled to a thickness of 0.590 inch, taking 10 percent reduction per pass and reheating after each pass. When a thickness of 1 inch was reached, the reheating temperature was progressively lowered; the final reductions were made at 1700 F.
- (4) U-212 steel, received as 2-15/16-inch-diameter bar stock 16 inches long. The bar was cut into two 8-inch lengths that were heated to 1925 F and forged to a thickness of 2-1/4 inches. These forged bars were reheated to 1925 F and hot rolled to a thickness of 0.525 inch, taking 10 percent reduction per pass and reheating after each pass.

The original lot of AISI Type 410 stainless steel that was received for evaluation did not respond in the normal manner to standard heat-treating procedures. A brief metallographic study of this material revealed that it contained considerable delta ferrite, a microconstituent not normally found in this alloy in appreciable amounts. Samples of the material were given various aging treatments in an attempt to reduce or eliminate the undesirable delta ferrite; however, these treatments were not successful. Therefore, it was concluded that this particular lot was not representative of the material and should not be used in this evaluation. Consequently, NASA agreed to furnish heat-treated and machined specimens of AISI Type 410 stainless steel for evaluation. These samples were not received until April, and, consequently, time did not permit the evaluation of this alloy to be completed.

Chemical Analyses

Samples of all the specified alloys were prepared for single-determination, wet-chemical analysis. In those cases where the value reported for a given element were significantly different from the vendor's certified analysis or were outside the specified chemical-composition limits, a second analysis was made for that element. The results of these analyses are listed in Table 2, along with the vendors' certified analyses. No analysis was received for the AISI Type 410 stainless steel provided by NASA, and because it was not received until late in the program, a check analysis was not conducted for this alloy.

Generally, the results of the check analyses were in good agreement with the vendors' certified analyses and, with one minor exception, were within the composition limits established for the respective alloys.

TABLE 1. ALLOYS UNDER STUDY AND THE STRENGTH LEVELS AT WHICH THEY WERE EVALUATED

Alloy	Ultimate Tensile Strength, psi
Ti-6Al-4V	160,000
AISI Type H-11 steel	260,000
AISI 4130 steel	180,000
AISI 8740 steel	180,000
18Ni maraging steel	260,000
Alloy 718 ^(a)	180,000
U-212 steel	180,000
René 41	200,000
Waspaloy	190,000 ^(b)
17-7 PH stainless steel	200,000 (RH 1025) ^(c)
17-4 PH stainless steel	200,000 (H-900) ^(d)
AM-355 stainless steel	180,000 (SCT 1000) ^(e)
AISI 4340 steel	260,000
AISI Type 410 stainless steel	180,000

(a) Alloy 718 is a nickel-base alloy that has the following nominal composition: 19Cr, 3Mo, 5Cb, 0.8Ti, 0.6Al, 18Fe, balance Ni. It was developed by the International Nickel Company and was first designated as Inconel 718. Other manufacturers licensed to produce the alloy have used other trade names, such as Lescalloy 718, Udimet 718, Allvac 718, FS-718, and Unitemp 718. There also may be other trade designations.

(b) Changed from 260,000-psi ultimate tensile strength.

(c) Changed from RH 1075.

(d) Changed from RH 900, 210,000-psi ultimate tensile strength.

(e) Changed from 190,000-psi ultimate tensile strength.

TABLE 2. CERTIFIED AND CHECK-ANALYSIS CHEMICAL COMPOSITIONS OF 13 OF THE ALLOYS UNDER INVESTIGATION

Identification	Material	Chemical Composition, weight percent											Other
		C	Mn	Si	P	S	Cr	Ni	Mo	Al	Fe		
A	AISI Type H-11 tool steel	Certified	0.41	0.36	0.99	0.015	0.002	4.99	--	1.26	--	Bal	0.48V, 0.09W
		Check	0.43	0.38	0.92	0.010	0.006	4.86	--	1.30	--	Bal	0.45V, 0.10W
B	AISI 4130 steel	Certified ^(a)	0.33	0.60	0.26	0.017	0.016	0.87	--	0.19	--	Bal	--
C	AISI 4340 steel	Certified ^(a)	0.41	0.83	0.30	0.010	0.018	0.85	1.83	0.25	--	Bal	--
		Check	0.068	0.56	0.38	0.016	0.017	17.22	7.24	--	1.20	Bal	--
D	17-7 PH stainless steel	Certified	0.10	0.69	0.39	0.025	0.013	17.40	7.22	--	1.26	Bal	--
		Check	0.037	0.32	0.69	0.017	0.005	15.67	4.35	--	--	Bal	3.56Cu, 0.21 Cb + Ta
E	17-4 PH stainless steel	Certified	0.05	0.38	0.69	0.018	0.007	15.64	4.20	--	--	Bal	3.46Cu, 0.32 Cb + Ta
		Check	0.04	--	--	--	--	--	--	6.27	0.08	4.12V, 0.013N, 0.112O (H = 42 ppm)	
F	AISI Type 410 stainless steel	Certified ^(d)	<0.03	--	--	--	--	--	--	5.97	0.11	4.11V, 0.015N	
G	AM-355 stainless steel	Certified	0.12	0.93	0.16	0.018	0.008	15.14	4.27	2.65	--	Bal	0.082N
		Check	0.13	0.96	0.16	0.020	0.010	15.27	4.18	2.64	--	Bal	0.082N
H	Ti-6Al-4V	Certified	0.04	--	--	--	--	--	--	--	6.27	0.08	4.12V, 0.013N, 0.112O (H = 42 ppm)
I	18Ni maraging steel (250)	Certified	0.005	0.02	0.020	0.003	0.005	--	18.48	4.90	0.07	Bal	7.51Co, 0.46Ti ^(c) , 0.0032B ^(b) , 0.016Zr ^(b) , >0.01Ca ^(b)
		Check	0.03	<0.01	0.01	0.003	0.006	--	18.39	5.07	0.07	Bal	7.43Co, 0.39Ti, 0.005Cu
J	Alloy 718	Certified	0.052	0.10	0.04	0.002	0.006	17.83	53.02	3.01	0.50	--	5.25 Cb + Ta, >0.05Cu, 0.17Co, 1.05Ti, 0.0054B
		Check	0.09	0.13	0.29	0.005	0.009	18.78	52.64	3.04	0.61	Bal	5.47 Cb + Ta, 0.008Cu, 0.22Co, 0.79Ti, 0.0028B
K	AISI E 8740 steel	Certified	0.405	0.87	0.28	0.007	0.010	0.54	0.56	0.23	--	Bal	--
		Check	0.41	0.92	0.24	0.008	0.013	0.62	0.52	0.23	--	Bal	0.0079B, 0.060Zr
L	Waspaloy	Certified	0.06	0.02	0.02	0.003	0.010	18.64	Bal	4.04	1.32	0.66	12.75Co, 2.88Ti, <0.06Cu, 0.0079B, 0.060Zr
		Check	0.08	0.008	<0.01	<0.005	0.008	19.19	Bal	4.21	1.46	1.06	12.79Co, 3.31Ti, <0.01Cu, 0.0055B, 0.08Zr

TABLE 2. (Continued)

Identification	Material	Chemical Composition, weight percent												
		C	Mn	Si	P	S	Cr	Ni	Mo	Al	Fe	Other		
M	René 41	Certified	0.082	0.05	0.030	0.006	0.006	0.006	18.50	Bal	9.70	1.55	0.88	10.75Co, <0.06Cu, 3.20Ti, 0.006B
		Check	0.09	0.01	0.05	<0.005	0.007	19.19	Bal	9.48	1.44	1.15	11.00Co, <0.01Cu, 2.60Ti, 0.005B	
N	U-212 steel	Certified	0.092	0.01	0.01	0.004	0.008	16.12	25.36	--	0.20	Bal	3.87Ti, 0.06B, 0.037Zr, 0.42 Cb-Ta	
		Check	0.10	Trace	<0.01	--	0.010	15.46	25.50	--	0.14	Bal	3.84Ti, 0.05B, 0.02Zr, 0.53Cb	

(a) Certified analysis not received.

(b) Added.

(c) Erroneously reported as 0.14 percent Ti in the November 12, 1965, quarterly report.

(d) Material received from NASA; no analyses provided.

Heat-Treating Studies

It was necessary to establish a heat treatment that would produce the specified strength levels in the various materials. For this purpose, eight bars, 1/2 x 1/2 x 3 inches, were cut from each material with the long dimension parallel to the primary rolling direction. These bars were rough machined into tensile specimens to be used to evaluate the selected heat treatments. Two bars of each alloy were given a selected heat treatment and then were finish machined to the dimensions of the standard 1/4-inch-diameter tensile specimen shown in Figure 3. The specimens were subjected to conventional tensile tests (platen speed, 0.02 in./min) to determine their mechanical properties. If necessary, the treatments were modified and additional specimens were treated and tested to verify the properties.

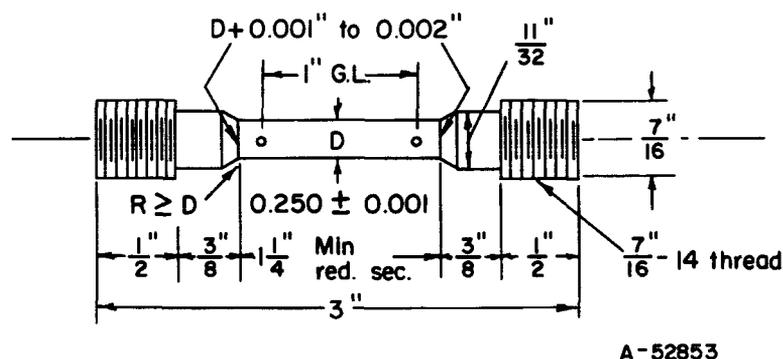


FIGURE 3. STANDARD 1/4-INCH-DIAMETER TENSILE SPECIMEN

The heat treatments established and the resultant mechanical properties for the respective alloys are listed in Table 3.

Sample Preparation

The sample configurations used in this study consisted of four types: (1) smooth tensile specimens, as shown in Figures 4 and 5; (2) notched tensile specimens, Figure 6; (3) hydrogen-entry-rate specimens, 3 x 3 x 0.250-inch plates, and (4) specimens for hydrogen analysis, 0.250 inch in diameter by approximately 1 inch in length.

The smooth and notched tensile specimens were prepared as follows: bars 1/2 x 1/2 x 6 inches were cut from each material with the long direction parallel to the primary working direction. These bars were rough machined and then heat treated according to the procedures established in the heat-treating studies. The heat treatments were verified by hardness checks. In those cases where the hardness varied from the value obtained in the heat-treating studies, tensile specimens were machined and tested to verify the properties. The specimens were then finish machined to the dimensions shown in Figure 4 or Figure 6. Subsequently, the reduced section of the smooth specimens was electropolished to a diameter of 0.250 inch in final preparation for testing. Twelve smooth tensile specimens were machined from each alloy, while 25 notched tensile specimens were machined from only those alloys found to be susceptible to hydrogen-stress cracking in the Phase I experiments. The specimens of AISI Type 410

TABLE 3. HEAT TREATMENTS AND MECHANICAL PROPERTIES OF THE ALLOYS INVESTIGATED

Identification	Material	Desired Strength Level, psi	Heat Treatment	Sample Number	Mechanical Properties				
					Ultimate Tensile Strength, psi	Yield Strength (0.2% Offset), psi	Elongation in 1 Inch, percent	Reduction of Area, percent	Rockwell C Hardness
A	AISI Type H-11 tool steel	260,000	Preheat at 1450 F for 30 min, austenitize at 1850 F for 30 min, air cool, double temper at 1075 F for 2 hr each temper, air cool between temper and after final temper.	A-7 A-8	268,500 266,000	230,200 227,600	13 15	43.5 48	51 --
B	AISI 4130 steel	180,000	Austenitize at 1550 F for 30 min, oil quench, temper at 850 F for 2 hr, air cool.	B-7 B-8	187,700 187,900	170,800 170,800	15 14	54.5 53.5	43 --
C	AISI 4340 steel	260,000	Austenitize at 1550 F for 30 min, oil quench, temper at 525 F for 2 hr, air cool.	C-3 C-4	261,100 263,000	223,900 224,000	12 12.5	54 54.5	50 --
D	17-7 PH stainless steel (RH 1025)	200,000	Austenitize at 1950 F for 30 min, air cool, condition at 1750 F for 30 min, air cool, refrigerate at -100 F for 8 hr, age at 1025 F for 1 hr, air cool.	D-7 D-8	202,600 204,000	158,500 162,600	18 18	35.5 40	41 --
E	17-4 PH stainless steel (H-900)	200,000	Austenitize at 1900 F for 30 min, air cool, age at 900 F for 1 hr, air cool.	E-1 E-2	203,500 203,900	177,800 177,900	19 20	53 54	41.5 --
F	AISI Type 410(a) stainless steel	180,000	Austenitize in vacuum furnace at 1775 F for 45 min, oil quench, temper in vacuum furnace at 575 F for 2 hr.	F-1(b) F-2(b) F-3(c)	186,300 188,500 190,000	143,800 143,800 145,800	10.5(d) 11.0(d) 8.0(d)	-- -- --	-- -- 43
G	AM-355 stainless steel (SCT 1000)	180,000	Austenitize at 1950 F for 30 min, water quench, condition at 1750 F for 45 min, water quench, refrigerate at -100 F for 3 hr, age at 1000 F for 3 hr, air cool.	G-5 G-6	179,400 178,700	163,700 163,500	21 22	61.5 61.5	43 --
H	Ti-6Al-4V	160,000 psi	Solution treat at 1550 F for 30 min, water quench, age at 900 F for 6 hr, air cool.	H-3 H-4	163,100 159,300	143,500 142,900	14 14	25.5 24.5	-- --
I	18Ni maraging steel (250)	260,000	Anneal at 1500 F for 1 hr, air cool, age at 900 F for 3 hr, air cool.	I-1 I-2	262,800 265,000	259,000 258,000	13 13	62 63	50 --
J	Alloy 718	180,000	Solution treat at 1800 F for 1 hr, air cool, age at 1325 F for 16 hr, air cool.	J-1 J-2	204,500 203,100	167,700 163,700	-- 26	43.5 39	45 --
K	AISI E 8740	180,000	Normalize at 1600 F for 30 min, air cool, austenitize at 1550 F for 30 min, oil quench, temper at 950 F for 2 hr, air cool.	K-1 K-2	182,600 181,700	172,600 170,900	16 17	56.5 57	43 --
L	Waspaloy	190,000	Solution treat at 1825 F for 4 hr, air cool, stabilize at 1550 F for 4 hr, air cool, age at 1400 F for 16 hr, air cool.	L-1 L-2	189,500 189,900	117,600 118,200	30 30	34.5 33.5	36.5 --
M	René 41	200,000	Solution treat at 1950 F for 4 hr, air cool, age at 1400 F for 16 hr, air cool.	M-1 M-2	198,100 199,300	134,000 133,300	29 26	25.5 24	42.5 --
N	U-212 steel	180,000	Solution treat at 1850 F for 2 hr, water quench, heat at 1425 F for 2 hr, air cool, age at 1250 F for 16 hr, air cool.	N-1 N-2	184,400 186,100	130,400 131,500	28 27.5	59.5 57.5	40 --

(a) Heat treated by NASA. (b) Flat specimen. (c) Round specimen. (d) Percent in 2 inches.

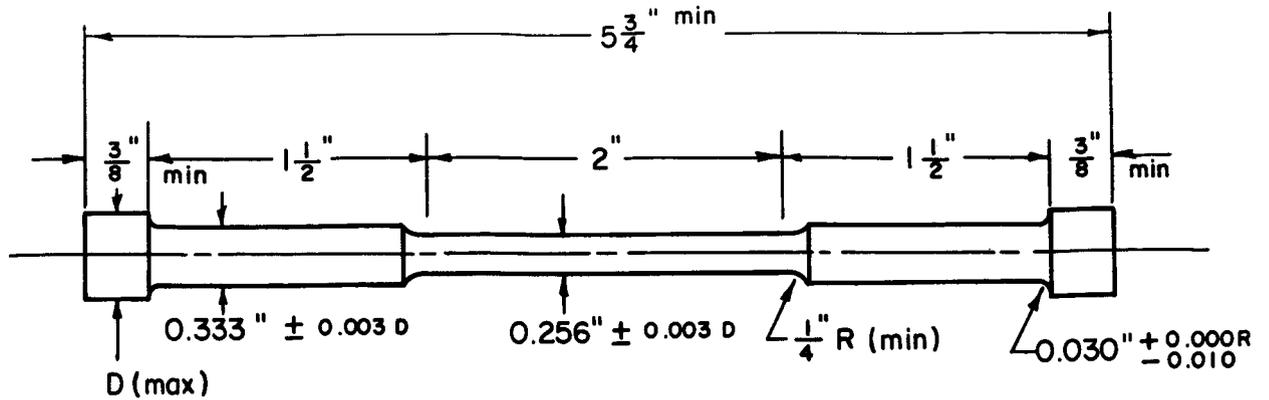


FIGURE 4. DETAILS OF THE UNNOTCHED TENSILE SPECIMENS USED IN THE HYDROGEN-EMBRITTELEMENT STUDIES (AS FINISHED MACHINED, PRIOR TO ELECTROPOLISHING)

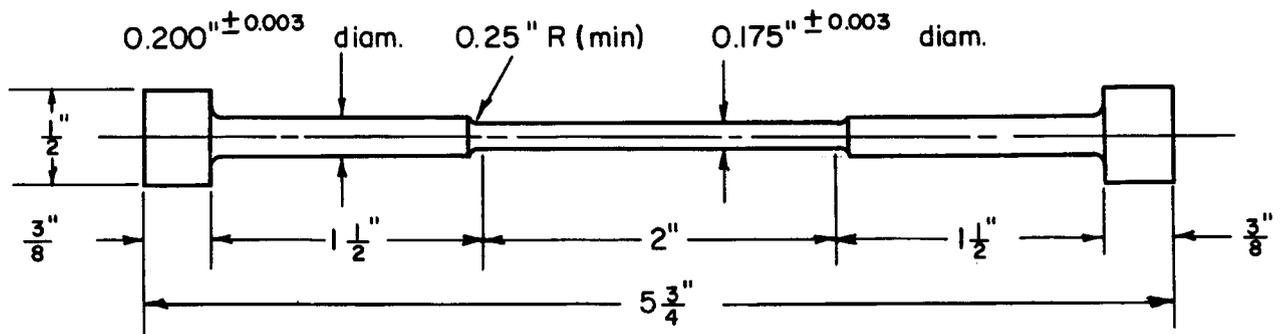


FIGURE 5. DETAILS OF UNNOTCHED TENSILE SPECIMENS USED TO EVALUATE THE SUSCEPTIBILITY OF AISI TYPE 410 STAINLESS STEEL TO HYDROGEN-STRESS CRACKING (AS FINISHED MACHINED, PRIOR TO ELECTROPOLISHING)

Machined from 1/4-inch plate.

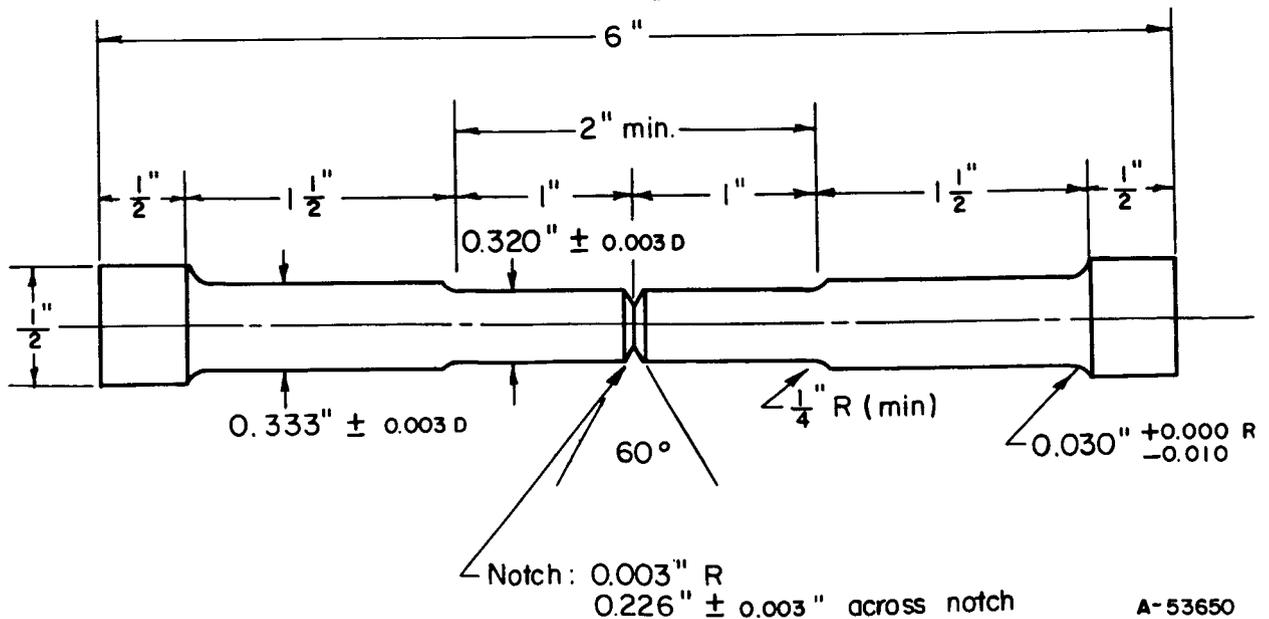


FIGURE 6. DETAILS OF THE NOTCHED TENSILE SPECIMEN USED IN THE HYDROGEN-EMBRITTELEMENT STUDIES

stainless steel provided by NASA were smaller, as shown in Figure 5. After electropolishing, the diameter of the reduced section of these specimens was about 0.168 inch.

The hydrogen-entry-rate specimens were prepared by cutting 3 x 3-inch plates from each material and rough machining equal amounts of material from each side to produce a thickness of 0.265 inch. The specimens were heat treated along with the tensile bars and then finish ground to a final thickness of 0.256 inch by removing equal amounts of material from both sides. Subsequently, they were electropolished to a thickness of approximately 0.250 inch.

The specimens for hydrogen analysis were cut from bars that were left over from the Phase 1 hydrogen-stress-cracking experiments. It was essential that these specimens have the same diameter (0.250 inch as electropolished and ready for testing) as the portions of the tensile specimens that would be cathodically charged, so that conditions for hydrogen entry and diffusion would be the same when prepared and charged under identical conditions.

EXPERIMENTAL PROCEDURES

Phase 1. Preliminary Study of the Susceptibilities of the Specified Alloys to Hydrogen-Stress Cracking

Hydrogen-Entry-Rate Experiments

The procedure used to determine the hydrogen-entry rate was as follows: the hydrogen-entry-rate specimen was degreased in trichloroethylene, and then one surface was electropolished to remove the cold-worked material that resulted from machining, and to provide a clean, bright surface. This electropolished surface was used as the entry surface. The specimen was masked off with electroplater's tape, leaving 0.8 square inch of the electropolished surface exposed. The specimen was then placed in the entry-rate apparatus and cathodically charged with hydrogen for 4 hours. Readings of bubble displacement were made for both the current-efficiency cell and the hydrogen-entry cell; these readings and the temperature and barometric pressure were taken at 10-minute intervals during the first hour of charging and at 20-minute intervals for the remaining 3 hours. Gas volumes were obtained for both cells by the difference in bubble positions in the calibrated tubes. Subtracting the gas volume in the hydrogen-entry cell (oxygen plus unabsorbed hydrogen) from the gas volume in the current-efficiency cell (total hydrogen plus oxygen liberated by electrolysis) for a given time interval gave the volume of hydrogen absorbed by the steel in that interval. The temperature and pressure readings were used to convert the gas volumes to standard conditions. The values for volume of absorbed hydrogen were then converted to weight in micrograms, and the results were plotted as weight of hydrogen occluded versus time of charging. The slopes of the plots corresponded to the rate at which hydrogen entered the specimens.

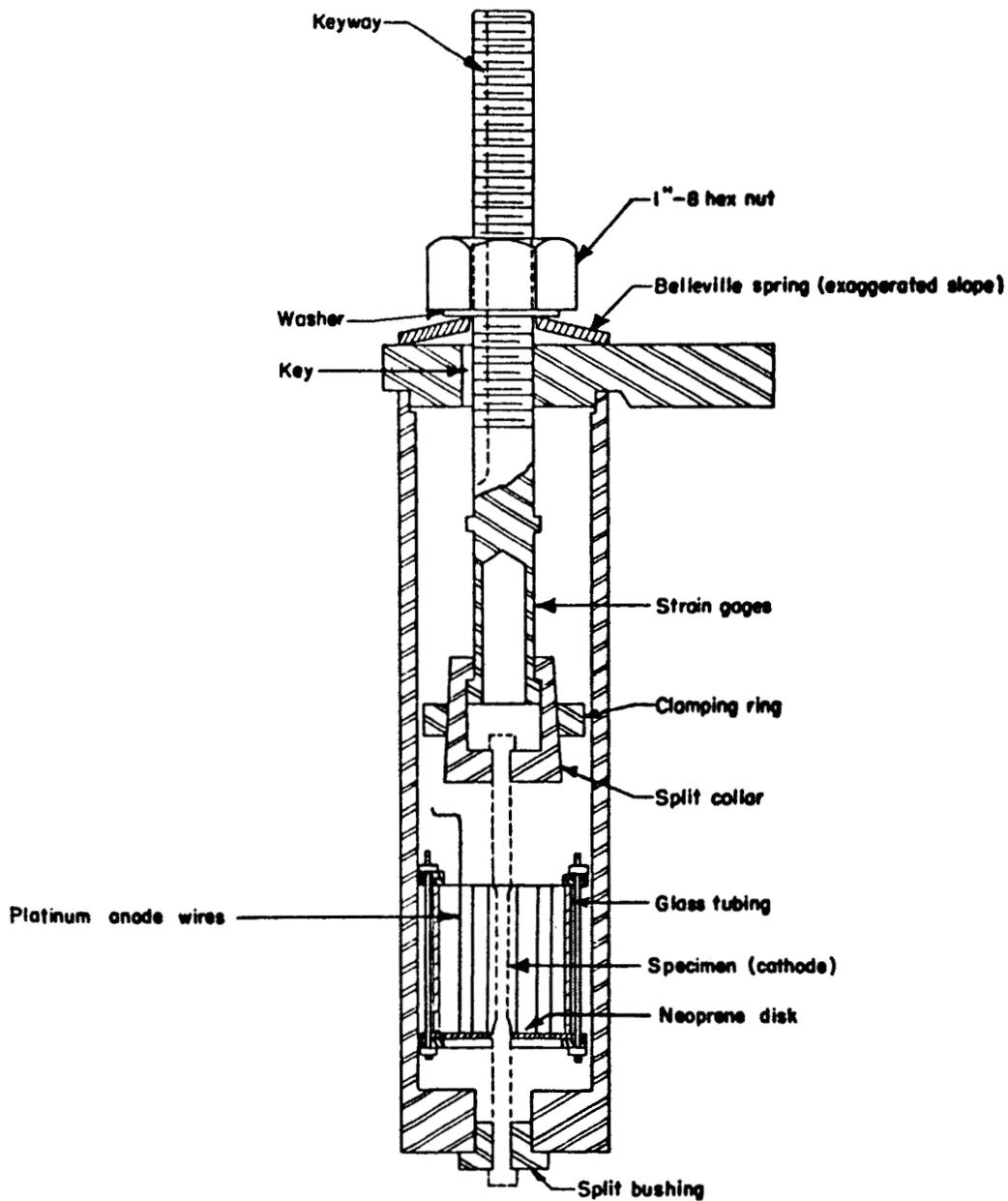
Hydrogen-Stress-Cracking Experiments

The following procedure was used to determine the susceptibility of the various materials to hydrogen-stress cracking. The smooth tensile specimens were degreased in trichloroethylene and electropolished to a diameter of 0.250 ± 0.002 inch to remove the cold-worked layer and to provide a smooth, clean surface. The electropolished surfaces were then lightly rubbed with Linde A alumina powder and alcohol to remove stains that sometimes were left by the electropolishing operation. The specimens were rinsed in alcohol and stored in a desiccator until they were used. Prior to performing the hydrogen-stress-cracking experiment, the specimens were masked off with electroplater's tape, leaving a 1-inch-long exposed length (approximately 0.8 square inch of exposed area) in the reduced section. The specimens were then placed in the sustained-tensile-stress apparatus shown in Figure 7, and the desired stress was applied by tightening the nut on the loading screw. The stress was monitored by strain gages (on the hollow portion of the loading screw) and an SR-4 strain indicator; the loading device was calibrated periodically in a universal testing machine. The desired cathodic charging condition was then imposed on the specimen. The sustained-load apparatus was connected through a microswitch to an automatic timer that recorded the time for failure.

Four charging conditions, which ranged from severe to very mild, were used to evaluate the sensitivity of the specified materials to hydrogen-stress cracking. These conditions were:

- (1) Condition A - severe charging condition.
 Electrolyte - 4% by weight of H_2SO_4 in distilled water + 5 drops per liter of cathodic poison consisting of 2 g phosphorus dissolved in 40 ml CS_2
 Current density - 8 ma/in.²
- (2) Condition B - medium charging condition.
 Electrolyte - 0.004% by weight of H_2SO_4 in distilled water + 5 drops per liter of cathodic poison composed of 2 g phosphorus dissolved in 40 ml CS_2
 Current Density - 0.625 ma/in.²
- (3) Condition C - mild charging condition.
 Electrolyte - 5 parts by volume lactic acid in ethylene glycol (the lactic acid reagent contained approximately 15 percent water by weight)
 Current density - 0.125 ma/in.²
- (4) Condition D - very mild charging condition.
 Electrolyte - 5 parts by volume lactic acid in ethylene glycol (the lactic acid reagent contained approximately 15 percent water by weight)
 Current density - 0.076 ma/in.².

Specimens of the specified alloys were initially charged under Condition A. If the specimens failed, new specimens were charged under the next-milder condition, Condition B, and so on, until a specimen of the material could withstand 200 hours of charging while subjected to a static tensile stress equal to 80 percent of its yield strength or until a specimen had failed under each of the charging conditions.



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FIGURE 7. APPARATUS EMPLOYED IN HYDROGEN-STRESS-CRACKING EXPERIMENTS

When cleaned and electroplated specimens were evaluated, the glass cell was removed, since no electrolyte was required.

Hydrogen Analyses

When the limiting conditions for hydrogen-stress cracking were determined, small samples of each of the alloys were charged under these same conditions and then were analyzed for hydrogen. The procedure used in these experiments was as follows: The specimens, which were cut from the reduced section of electropolished tensile bars, were cleaned in acetone and rinsed in alcohol. Then one specimen was charged under the mildest condition that produced a failure for the time it took to produce the failure in the hydrogen-stress-cracking experiment. Another specimen was charged under the most severe condition that did not produce a failure for approximately 200 hours. After charging, the specimens were immediately placed in liquid nitrogen and stored until analyzed. The average hydrogen content was determined by the tin-fusion vacuum-fusion technique.

Phase 2. Study of Susceptibility to Embrittlement Resulting From Surface Treatments

Cleaning and Electroplating Procedures

Notched tensile specimens of AISI Type H-11 tool steel, AISI 4130, AISI 4340, AISI E 8740, and 18Ni maraging steel were evaluated for susceptibility to hydrogen-stress cracking after being cleaned and electroplated in conventional cadmium cyanide baths with and without brighteners. The bright-cadmium plating was done at a commercial plating facility, while the dull-cadmium plating was done in the laboratory using baths of the same composition - except for the plating bath, which contained no brightener. The procedures used were as follows:

- (1) Specimens degreased in acetone.
- (2) Anodic clean for 3 min in alkaline bath; 8 oz/gal cleaner (Pennsalt 78), temperature 180 F, current density 40 a/ft².
- (3) Double water rinse.
- (4) Pickle 10 sec in 50 percent by volume HCl with inhibitor (Rodine 60). The 18Ni maraging steel was reverse-current etched in 25 percent by weight H₂SO₄ for 10 sec anodic, 3 sec cathodic, instead of being pickled in the HCl solution.
- (5) Double water rinse. Part of the specimens were removed after this step for static loading and hydrogen analysis in the as-cleaned condition.
- (6) Cadmium plate in conventional cyanide cadmium bath for 10-12 min at 25 a/ft² current density. Bath temperature 80-90 F. Plate thickness 0.4 to 0.6 mil.
- (7) Double rinse in water.
- (8) Immerse in chromate conversion bath (Duracoat) for 10 sec.
- (9) Double rinse in water.

Both the as-cleaned and the cadmium-plated specimens were dried and then stored in liquid nitrogen until tested so as to prevent loss of hydrogen.

Notched tensile specimens of 17-7 PH stainless steel, 17-4 PH stainless steel, and AM-355 stainless steel were evaluated for susceptibility to hydrogen-stress cracking after cleaning and electroplating in a Wood's-nickel-strike bath. This work was done at a commercial plating facility using the following procedure:

- (1) Specimens were degreased in trichloroethylene.
- (2) Anodic clean for 1 min.
- (3) Water rinse.
- (4) Pickle 10 sec in 50 percent by volume HCl.
- (5) Water rinse.
- (6) Reverse current etch in 25 percent by weight H_2SO_4 .
- (7) Water rinse. Part of the samples were removed after this treatment for static loading and hydrogen analyses.
- (8) Nickel strike for 5 min, all-chloride bath, current density 100 a/ft².
- (9) Water rinse.

The specimens were dried in an air blast and stored in liquid nitrogen until used.

Hydrogen-Stress-Cracking Experiments

The cleaned and electroplated notched tensile specimens were loaded into the hydrogen-stress-cracking apparatus shown in Figure 7 as follows:

- (1) Cleaned specimens - loaded to 90 percent of the average notched tensile strength of the respective alloys. If the specimens did not fail in 100 hr, they were considered to be nonembrittled and were removed from the apparatus.
- (2) Electroplated specimens - loaded to 75 percent of the average notched tensile strength of the respective alloys. If the specimens did not fail in 100 hr, the applied stress was increased to 90 percent of the notched tensile strength. If the specimens did not fail in another 100 hr, they were considered to be nonembrittled and were removed from the apparatus. In some instances where two specimens of an alloy withstood a stress of 75 percent of the notched tensile strength but failed at 90 percent, a third specimen was loaded directly to 90 percent.

Hydrogen Analyses

Small samples of each of the alloys were subjected to the cleaning and electroplating treatments and then were analyzed for average hydrogen content using the tin-fusion vacuum-fusion technique. These specimens were stored in liquid nitrogen to prevent loss of hydrogen prior to analysis.

RESULTS AND DISCUSSION

Phase 1. Preliminary Investigation of the Susceptibilities of the Specified Alloys to Hydrogen Embrittlement

The results of the Phase 1 experiments are presented in Tables 4, 5, and 6.

Table 4 presents the hydrogen-stress-cracking data. The results obtained indicate that the alloys exhibit significant differences in susceptibility to hydrogen-stress cracking. Five of the alloys (Ti-6Al-4V, Alloy 718, Waspaloy, René 41, and U-212 steel) were found to be insensitive to hydrogen-stress cracking under the most severe charging condition used. All the other alloys failed in times ranging from a few minutes to about 6 hours under this charging condition. As the severity of the charging condition was reduced, the differences in the relative susceptibilities of the alloys became more apparent. For purposes of comparison, the alloys have been grouped as follows (tensile-strength levels in parentheses):

Group 1. Not susceptible to failure in 200 hours under the most severe charging condition

Ti-6Al-4V	(160,000 psi)
Alloy 718	(180,000 psi)
Waspaloy	(190,000 psi)
René 41	(200,000 psi)
U-212 steel	(180,000 psi)

Group 2. Failed under only the most severe charging condition (Condition A)

17-7 PH stainless steel (200,000 psi)

Group 3. Failed under Conditions A and B

AM-355 stainless steel	(180,000 psi)
18Ni maraging steel	(260,000 psi)
AISI E 8740 steel	(180,000 psi)

Group 4. Failed under Conditions A, B, and C

AISI Type H-11 tool steel	(260,000 psi)
17-4 PH stainless steel	(200,000 psi)

TABLE 4. RESULTS OF HYDROGEN-STRESS-CRACKING EXPERIMENTS

Sample	Yield-Strength Level (0.2% Offset), psi	Charging Condition	Applied Stress ^(a) , psi	Time for Failure, hr
<u>AISI Type H-11 Steel</u>				
A-1	229,000	A	185,000	4.6
A-2	229,000	A	185,000	6.1
A-3	229,000	A	185,000	5.5
A-4	229,000	B	185,000	18.4
A-5	229,000	C	185,000	98.5
A-6	229,000	D	185,000	>215.3 ^(b)
A-7	229,000	D	185,000	>356.7 ^(b)
<u>AISI 4130 Steel</u>				
B-1	170,000	A	135,000	0.4
B-2	170,000	A	135,000	0.5
B-3	170,000	A	135,000	0.6
B-4	170,000	B	135,000	1.0
B-5	170,000	C	135,000	32.4
B-6	170,000	D	135,000	36.8
<u>AISI 4340 Steel</u>				
C-1	225,000	A	180,000	0.05
C-2	225,000	A	180,000	0.05
C-3	225,000	B	180,000	0.6
C-4	225,000	C	180,000	9.5
C-5	225,000	D	180,000	9.2
<u>17-7 PH Stainless Steel</u>				
D-1	160,000	A	128,000	1.5
D-2	160,000	A	128,000	0.6
D-3	160,000	A	128,000	1.2
D-4	160,000	B	128,000	>273.1 ^(b)
D-5	160,000	B	128,000	>216.3 ^(b)
<u>17-4 PH Stainless Steel</u>				
E-1	180,000	A	160,000 ^(c)	0.3
E-2	180,000	A	140,000	1.6
E-3	180,000	A	140,000	1.6
E-4	180,000	B	140,000	12.3
E-5	180,000	C	140,000	86.1
E-6	180,000	D	140,000	>215.9 ^(b)
E-7	180,000	D	140,000	>263.1 ^(b)
<u>AISI Type 410 Stainless Steel</u>				
F-5	145,000	A	116,000	1.2
F-6	145,000	A	116,000	1.7
F-7	145,000	A	116,000	1.6
F-4	145,000	B	116,000	>211.0 ^(b)
F-8	145,000	B	116,000	105.3

TABLE 4. (Continued)

Sample	Yield-Strength Level (0.2% Offset), psi	Charging Condition	Applied Stress ^(a) , psi	Time for Failure, hr
<u>AM-355 Stainless Steel</u>				
G-2	160,000	A	128,000	3.9
G-3	160,000	A	128,000	5.0
G-4	160,000	A	128,000	2.9
G-5	160,000	B	128,000	154.0
G-6	160,000	C	128,000	>235.2 ^(b)
G-7	160,000	C	128,000	>210.2 ^(b)
<u>Ti-6Al-4V</u>				
H-1	143,000	A	115,000	>264.0 ^(b)
H-2	143,000	A	115,000	>216.0 ^(b)
<u>18Ni Maraging Steel</u>				
I-1	258,000	A	200,000	5.1
I-2	258,000	A	200,000	5.8
I-3	258,000	A	200,000	5.0
I-4	258,000	B	200,000	21.4
I-5	258,000	C	200,000	>215.4 ^(b)
I-6	258,000	C	200,000	>215.9 ^(b)
<u>Alloy 718</u>				
J-1	162,000	A	130,000	>217.0 ^(b)
J-2	162,000	A	130,000	>213.9 ^(b)
<u>AISI E 8740 Steel</u>				
K-1	170,000	A	135,000	0.3
K-2	170,000	A	135,000	0.4
K-3	170,000	B	135,000	5.6
K-4	170,000	C	135,000	>309.2 ^(b)
K-5	170,000	C	135,000	>235.7 ^(b)
<u>Waspaloy</u>				
L-1	118,000	A	95,000	>263.3 ^(b)
L-3	118,000	A	95,000	>231.9 ^(b)
<u>René 41</u>				
M-1	134,000	A	107,000	>210.3 ^(b)
M-2	134,000	A	107,000	>238.3 ^(b)
<u>U-212 Steel</u>				
N-1	131,000	A	105,000	>209.1 ^(b)
N-2	131,000	A	105,000	>202.8 ^(b)

(a) Applied stress is approximately 80 percent of the yield strength, except for one specimen as noted.

(b) Runout; specimen did not fail.

(c) Approximately 89 percent of the nominal yield strength.

TABLE 5. RESULTS OF HYDROGEN-ENTRY-RATE EXPERIMENTS
(Charging Condition A)

Sample Number	Material	Hydrogen-Entry Rate During First Hour of Charging, $\mu\text{g}/\text{in.}^2\text{-min}$	
		Individual Values	Average
A-1	AISI Type H-11 tool steel	1.41	1.26
A-2	AISI Type H-11 tool steel	1.11	
B-1	AISI 4130 steel	0.85	0.79
B-2	AISI 4130 steel	0.72	
C-1	AISI 4340 steel	0.58	0.58
D-1	17-7 PH stainless steel	1.02	0.81
D-2	17-7 PH stainless steel	0.59	
E-1	17-4 PH stainless steel	1.37	1.59
E-2	17-4 PH stainless steel	1.82	
F-1	AISI Type 410 stainless steel	0.77	0.61
F-2	AISI Type 410 stainless steel	0.45	
G-1	AM-355 stainless steel	1.04	0.95
G-2	AM-355 stainless steel	0.85	
H-1	Ti-6Al-4V	1.97	2.27
H-2	Ti-6Al-4V	2.56	
I-1	18Ni maraging steel	0.98	0.97
I-2	18Ni maraging steel	0.96	
J-1	Alloy 718	0.63	0.56
J-2	Alloy 718	0.49	
K-1	AISI E 8740 steel	1.07	0.97
K-2	AISI E 8740 steel	0.87	
K-3	AISI E 8740 steel	0.92 ^(a)	0.96
K-4	AISI E 8740 steel	1.00 ^(a)	
L-1	Waspaloy	0.28	0.38
L-2	Waspaloy	0.47	
M-1	René 41	0.21	0.21
N-1	U-212 steel	0.25	0.30
N-2	U-212 steel	0.35	

(a) Duplicate experiments conducted to check renovated apparatus.

TABLE 6. RESULTS OF HYDROGEN ANALYSES PERFORMED ON SPECIMENS OF THE SPECIFIED ALLOYS CATHODICALLY CHARGED UNDER THE LIMITING CONDITIONS FOR HYDROGEN-STRESS CRACKING

Sample Number	Material	Charging Condition	Charging Time, hr	Average Hydrogen Content, ppm by weight (± 0.1)
A-1	AISI Type H-11 tool steel	D	221.4	6.6
A-2	AISI Type H-11 tool steel	C	98.5	4.4
B-1	AISI 4130 steel	D	36.8	6.0
C-1	AISI 4340 steel	D	9.2	0.9
D-2	17-7 PH stainless steel	B	201.4	5.3
D-1	17-7 PH stainless steel	A	1.5	6.5
E-1	17-4 PH stainless steel	D	212.4	3.4
E-2	17-4 PH stainless steel	C	88.0	2.7
G-2	AM-355 stainless steel	C	201.4	8.8
G-1	AM-355 stainless steel	B	154.0	6.7
H-1	Ti-6Al-4V	A	201.5	3.0
I-1	18Ni maraging steel	C	212.2	1.6
I-2	18Ni maraging steel	B	21.4	3.4
J-1	Alloy 718	A	201.6	43.7
K-1	AISI E 8740 steel	C	212.2	2.2
K-2	AISI E 8740 steel	B	5.6	3.2
L-1	Waspaloy	A	201.6	17.4
M-1	René 41	A	201.6	34.1
N-1	U-212	A	201.6	13.7

Group 5. Failed under all conditions (A, B, C, D)

AISI 4340 steel	(260,000 psi)
AISI 4130 steel	(180,000 psi)

The evaluation of AISI Type 410 stainless steel was not completed, but it was shown to be susceptible to failure under at least Conditions A and B.

Four of the five alloys in Group 1 (the nickel-base alloys Waspaloy, Alloy 718, and René 41, and the austenitic U-212 steel) have a face-centered cubic structure. As was pointed out in the literature and industrial survey⁽⁹⁾, face-centered cubic alloys have been shown generally to be insensitive to hydrogen-stress cracking at room temperature. It is interesting to note that these four alloys exhibited the lowest hydrogen-entry rates (Table 5) but had the highest hydrogen contents (after charging for 200 hours under the severe conditions that did not produce failure in the statically loaded specimen), as shown in Table 6. The higher hydrogen contents are attributed to higher solubility of hydrogen in the face-centered cubic lattice and the long-time charging. The lower entry rates may be explained by the low diffusion rate of hydrogen in face-centered cubic alloys. Presumably, the surface layer soon became essentially saturated with hydrogen because hydrogen could not readily diffuse into the bulk of the material, and, therefore, these materials could no longer accept hydrogen at an appreciable rate. However, hydrogen gradually diffusing inward over the long-time period (200 hours) employed in the charging of these four materials allowed the average hydrogen content to build up to a high level because of the higher hydrogen solubility of these alloys.

The other alloy in this group of insensitive materials was Ti-6Al-4V. Other investigators have shown that it is susceptible to hydrogen embrittlement, but the hydrogen tolerance has been estimated to be approximately 200 ppm. Presumably, during these experiments insufficient hydrogen was picked up by this alloy to produce cracking. However, whether this was the result of unfavorable cell conditions or an inherent characteristic of titanium alloys under cathodic charging was not determined.

In view of the fact that other investigators have shown this alloy to be susceptible to hydrogen embrittlement under conditions where large amounts of hydrogen have been introduced, evaluation of this material will be continued in the next term of the contract. In this work, the electrolyte used will be one that is normally used in the etching or pickling of titanium.

Of the nine alloys that were susceptible to hydrogen-stress cracking, AISI 4340 steel appears to be the most susceptible, as it failed in the shortest times under all the cathodic charging conditions. The 17-7 PH stainless steel appears to be the least susceptible of these alloys; it failed only under the most severe cathodic charging condition (Condition A).

On the basis of the few experiments on hydrogen-entry rate, and for the conditions of heat treatment, surface preparation, and charging that were used, the alloys can be arranged in order of increasing hydrogen-entry rate as follows:

Insensitive Alloys

René 41
 U-212 steel
 Waspaloy
 Alloy 718
 Ti-6Al-4V

Sensitive Alloys

AISI 4340 steel
 AISI Type 410 stainless steel
 AISI 4130 steel
 17-7 PH stainless steel
 AM-355 stainless steel
 18Ni maraging steel
 AISI E 8740 steel
 AISI Type H-11 tool steel
 17-4 PH stainless steel

The entry rate observed for Ti-6Al-4V was 11 times that obtained for René 41.

The results of the hydrogen-entry-rate experiments suggest that there is no simple relationship between hydrogen-entry rate and susceptibility to hydrogen-stress cracking. For example, the most sensitive material (AISI 4340 steel) had the lowest hydrogen-entry rate of the nine alloys susceptible to hydrogen-stress cracking. However, the rate for AISI 4340 steel was almost the same as that found for Alloy 718, one of the insensitive materials. On the other hand, for the eight materials susceptible to hydrogen-stress cracking*, there was partial correlation between entry rate and relative susceptibility, as shown by the following tabulation:

<u>Material</u>	<u>Hydrogen-Entry Rate During First Hour of Charging, $\mu\text{g}/\text{in.}^2\text{-min}$</u>	<u>Relative Sensitivity to Hydrogen-Stress Cracking</u>
AISI 4340 steel	0.58	Group 5; failed under all charging conditions (A, B, C, D), even the very mild Condition D
AISI 4130 steel	0.79	
17-7 PH stainless steel	0.81	Group 2; failed only under Condition A (severe)
AM-355 stainless steel	0.95	Group 3; failed under Conditions A and B
18Ni maraging steel	0.97	
AISI E 8740 steel	0.97	
AISI Type H-11 tool steel	1.26	Group 4; failed under Conditions A, B, and C
17-4 PH stainless steel	1.59	

*Omitting AISI 410 stainless steel, for which the hydrogen-stress-cracking evaluation was incomplete.

Except for the AISI 4340 and AISI 4130 steels, which seemed to exhibit abnormally low entry rates, the susceptibility to hydrogen-stress cracking increased with increasing entry rate. The observed behavior of the AISI 4340 and AISI 4130 steels suggests that the critical hydrogen content to initiate failure may be more important than is hydrogen-entry rate.

For the eight sensitive materials for which the evaluation was complete, the correlation between average hydrogen content under limiting charging conditions for hydrogen-stress cracking and sensitivity to cracking was not so good as the relationships between cracking and entry rate. This is shown in the following tabulation:

Material	Average Hydrogen Content, ppm		Relative Sensitivity to Hydrogen-Stress Cracking	
	Under Most Severe Condition that did not Produce Failure	Under Mildest Condition that Produced Failure	Group	Mildest Charging Conditions Resulting in Failure
AISI 4340 steel	--	0.9	5	D, very mild
18Ni maraging steel	1.6	3.4	3	B, medium
AISI E 8740 steel	2.2	3.2	3	B, medium
17-4 PH stainless steel	3.4	2.7	4	C, mild
17-7 PH stainless steel	5.3	6.5	2	A, severe
AISI 4130 steel	--	6.0	5	D, very mild
AISI Type H-11 tool steel	6.6	4.4	4	C, mild
AM-355 stainless steel	8.8	6.7	3	B, medium

These data are added evidence that the critical hydrogen content to initiate failure is a very important factor in hydrogen-stress cracking.

For three of the steels (17-4 PH, H-11, and AM-355), the average hydrogen content for the mildest condition that produced failure was less than for the most severe condition that did not. At first glance, this might seem to be contradictory. However, the specimens that failed did so in less than 200 hours, while those that did not fail were under test for the full 200 hours. Charging for a long time at a low rate may result in a higher average hydrogen content than charging at a higher rate for a short time, but the hydrogen content near the surface of the specimen (where fracture starts) may be more closely related to the severity of the charging condition.

It is apparent from the results of the Phase 1 experiments that several factors are important in determining the susceptibility of materials to hydrogen-stress cracking. Among these factors are strength level, chemical composition, heat treatment (microstructure), and hydrogen content in the region of high tensile stress. For example, the three alloys heat treated to the 260,000 psi tensile-strength level (AISI 4340 steel, AISI Type H-11 tool steel, and 18Ni maraging steel) showed marked differences in their susceptibilities to failure.

The AISI 4340 steel is a low-alloy, high-strength, quenched-and-tempered conventional martensitic steel (that is, the martensite is iron-carbon martensite). AISI Type H-11 tool steel has a medium alloy content and is an air-hardening conventional martensitic steel. The 18Ni maraging steel is a high-alloy steel that is strengthened

by the precipitation of intermetallics in a special martensitic matrix (iron-nickel martensite). Of these three alloys, the 18Ni maraging steel was the least susceptible to hydrogen-stress cracking, while the AISI 4340 was the most susceptible. The major alloy additions in the maraging steel are nickel, cobalt, and molybdenum, while in the AISI Type H-11 tool steel, chromium, molybdenum, vanadium, and silicon are the major alloying elements. Several investigations have shown that alloy additions of silicon and chromium are particularly effective in decreasing the diffusivity of hydrogen in the body-centered cubic α -iron. The effect of these elements can partially explain the observed differences in the susceptibilities of these alloys to hydrogen-stress cracking. However, the effect on diffusivity does not explain the low hydrogen content in AISI 4340 (lowest Cr and Si contents of the three) under the mildest conditions that produced failure. The hydrogen contents were related, in part, to the charging times as follows:

AISI 4340 steel	9.2 hours	0.9 ppm
18Ni maraging steel	21.4 hours	3.4 ppm
AISI H-11 tool steel	98.5 hours	4.4 ppm

Other investigations have shown that AISI Type H-11 tool steel and 18Ni maraging steel are less susceptible to hydrogen-stress cracking than is AISI 4340 steel. Therefore, the results of this investigation are consistent with the previously observed behavior of these alloys.

Similarly for the 17-4 PH and 17-7 PH stainless steels heat treated to the 200,000-psi tensile-strength range, the observed differences in the susceptibilities of these alloys to hydrogen-stress cracking must be explained in terms other than strength level. The fracture surfaces of the 17-7 PH stainless steel specimens showed evidence of δ -ferrite banding in the alloy. Possibly, these bands acted as barriers to hydrogen diffusion, or they acted as crack arresters, or both. Because of the strength-level effect, bands of a lower strength material can be expected to resist hydrogen-stress cracking.

The steels heat treated to a strength level of 180,000 psi (which included AISI 4130 steel, AISI E 8740 steel, AM-355 stainless steel, and AISI Type 410 stainless steel) again showed differences in susceptibility to hydrogen-stress cracking based on the times for failure under the various charging conditions. Of these alloys, the AISI 4130 steel was the most susceptible to hydrogen-stress cracking, as it failed in relatively short times under all the charging conditions used in this evaluation. The high chromium content, which reduces hydrogen diffusivity, would explain at least partially why the AM-355 stainless steel was more resistant to hydrogen-stress cracking than was the low-alloy AISI 4130 steel. However, the composition fails to explain why the AISI E 8740 steel was far more resistant to hydrogen-stress cracking than was the AISI 4130 steel, since both have virtually the same chromium and silicon contents.

The results of the hydrogen analyses of specimens charged under the limiting hydrogen-stress-cracking conditions indicated that the various alloys have different hydrogen tolerance levels, i. e., the alloys exhibit differences in the amount of hydrogen required to initiate failure. However, several of the alloys charged under the conditions that did not produce failure contained more hydrogen than similar specimens charged under the somewhat more severe conditions that did result in failure. In some instances, this undoubtedly reflects the differences in charging time (longer times under the milder conditions). This behavior suggests that hydrogen distribution within the sample, rather than total hydrogen content, is more important in determining the susceptibility of a part to failure. It is rather generally agreed among various

investigators that under an applied stress hydrogen will diffuse to the region of maximum triaxial stress; when the hydrogen content in this region exceeds a critical level, it reacts with the material in an as yet incompletely understood way and initiates a crack. Consequently, the hydrogen content in the region of crack initiation may be significantly greater than the average hydrogen content of the sample, and as yet there is no method to determine hydrogen distribution within a sample that will provide knowledge of hydrogen content at specific sites.

Phase 2. Study of Susceptibility to Hydrogen-Stress
Cracking Resulting From Surface Treatments

In this phase of the research program, those alloys that were found to be susceptible to hydrogen-stress cracking in Phase 1 were evaluated to determine the degree to which they were embrittled by hydrogen introduced during conventional cleaning and electroplating processes. Five of the alloys (AISI Type H-11 tool steel, AISI 4130 steel, AISI 4340 steel, AISI E 8740 steel, and 18Ni maraging steel) were evaluated after cleaning and electroplating in cadmium cyanide baths with and without brighteners. Three of the stainless steels (17-4 PH, 17-7 PH, and AM-355) were evaluated after cleaning and electroplating in a Wood's-nickel-strike bath. The AISI Type 410 stainless steel was not received until late in the program, and, consequently, time did not permit evaluation of this alloy in the Phase 2 experiments.

The embrittling tendencies of the cleaning and electroplating processes were evaluated by subjecting notched tensile specimens of the various alloys to applied tensile stresses of 75 and/or 90 percent of their respective notched tensile strengths, as was previously described.

The notched tensile strength of each alloy was determined by loading specimens to failure in tension in a universal testing machine at a platen speed of 0.06 inch per minute. The average values of notched tensile strength obtained from at least two specimens in the as-heat-treated condition are listed in Table 7.

TABLE 7. AVERAGE NOTCHED TENSILE STRENGTHS OF THE
SUSCEPTIBLE ALLOYS AS HEAT TREATED

Identification	Material	Average Notched Tensile Strength, psi
A	AISI Type H-11 tool steel	306,000
B	AISI 4130 steel	265,000
C	AISI 4340 steel	324,000
D	17-7 PH stainless steel	199,000
E	17-4 PH stainless steel	306,000
G	AM-355 stainless steel	268,000
I	18Ni maraging steel	406,000
K	AISI E 8740 steel	273,000

The results of the hydrogen-stress-cracking experiments employing notched tensile specimens of the alloys that were cleaned and cadmium plated are listed in Table 8. The results of hydrogen analyses of samples of these alloys which had received the same processing are listed in Table 9.

The hydrogen-stress-cracking experiments indicated that the conventional preplating cleaning cycle performed at a commercial plating facility introduced sufficient hydrogen into several of the alloys to cause hydrogen-stress cracking. None of the samples that were cleaned in the laboratory, using baths of the same composition and the same procedures, indicated embrittlement. This behavior is more disturbing since the hydrogen analyses indicated that the laboratory cleaning generally introduced more hydrogen than did the commercial cleaning.

The results of the hydrogen-stress-cracking evaluations of the cadmium-plated specimens indicated that both the bright-cadmium plating bath and the dull-cadmium plating bath (same bath composition but without a brightener) caused hydrogen-stress cracking in certain of the alloys. However, the bright-cadmium bath was more embrittling than was the dull-cadmium bath, even though the dull-cadmium process introduced more hydrogen into the specimens. This behavior is attributed to the fact that the dull-cadmium plate is less dense (more porous) than is the bright-cadmium plate; therefore, it presented less of a barrier to hydrogen during the plating operation. Also, because it was porous, the dull-cadmium plate allowed sufficient hydrogen to effuse from the specimens during the static-loading experiments and before the failure mechanism became operative so that fracture did not initiate. Also, the throwing power of the dull-cadmium bath was not so great as that of the bright-cadmium bath; examination of some of the unbroken dull-cadmium-plated specimens after they were removed from the static-load apparatus showed incomplete plate coverage at the base of the notch.

Of the five alloys evaluated in these experiments, the AISI 4340 steel was the most severely embrittled, while the 18Ni maraging steel was the least embrittled. The fact that the three bright-cadmium-plated specimens of AISI E 8740 steel failed at the button end rather than at the notch suggests that pure axial loading was not achieved on these specimens, with the result that the maximum stress state occurred at the button end. This could have been caused by misalignment in the apparatus or, more likely, by warpage in the specimens.

The results of hydrogen analyses conducted on specimens of the various alloys after cleaning and electroplating showed that, in general, the plating process itself introduced more hydrogen into the specimens than did the preplating cleaning processes. The other results mentioned previously are that the dull-cadmium-plating bath introduced more hydrogen than did the bright-cadmium bath and that the laboratory cleaning cycle introduced more hydrogen than did the commercial plating cycle.

A comparison of the hydrogen contents of the materials after cleaning and cadmium plating with those after cathodic charging under the limiting conditions for hydrogen-stress cracking shows that in only the AISI 4340 steel was the hydrogen content greater after cleaning and electroplating than after cathodic charging. This observation is generally consistent with the results of the hydrogen-stress-cracking data, as the AISI 4340 steel was the most severely embrittled by the cleaning and electroplating procedures.

TABLE 8. RESULTS OF HYDROGEN-STRESS-CRACKING EXPERIMENTS
EMPLOYING CLEANED AND CADMIUM-PLATED SPECIMENS

Sample Number	Condition	Applied Stress, percent of NTS ^(a)	Time for Failure, hr
<u>AISI Type H-11 Tool Steel, NTS = 306,000 psi</u>			
A-8	Cleaned in laboratory	90	>100(b)
A-1	Bright-Cd plated	75	>118(b)
		90	(c)
A-3	Bright-Cd plated	75	>100(b)
		90	32.4
A-4	Bright-Cd plated	90	(c)
A-5	Bright-Cd plated	90	>117(b)
A-9	Dull-Cd plated	75	>120(b)
		90	>100(b)
A-10	Dull-Cd plated	75	>100(b)
		90	>176(b)
<u>AISI 4130 Steel, NTS = 265,000 psi</u>			
B-1	Cleaned commercially	90	6.6
B-8	Cleaned in laboratory	90	>116(b)
B-2	Bright-Cd plated	75	>118(b)
		90	2.9
B-3	Bright-Cd plated	75	>113(b)
		90	24.1
B-4	Bright-Cd plated	90	3.1
B-9	Dull-Cd plated	75	>137(b)
		90	1.2
B-12	Dull-Cd plated	75	>115(b)
		90	>147(b)
B-7	Dull-Cd plated	90	(c)
<u>AISI 4340 Steel, NTS = 324,000 psi</u>			
C-1	Cleaned commercially	90	(c)
C-3	Cleaned commercially	90	(c)
C-7	Cleaned in laboratory	90	>186(b)
C-11	Cleaned in laboratory	90	>100(b)
C-2	Bright-Cd plated	75	(c)
C-4	Bright-Cd plated	75	>0.05
C-5	Bright-Cd plated	75	>0.05
C-9	Dull-Cd plated	75	5.2
C-12	Dull-Cd plated	75	3.0

TABLE 8. (Continued)

Sample Number	Condition	Applied Stress, percent of NTS(a)	Time for Failure, hr
<u>18Ni Maraging Steel, NTS = 406,000 psi</u>			
I-1	Cleaned commercially	90	>229(b)
I-12	Cleaned in laboratory	90	>191(b)
I-3	Bright-Cd plated	75	>118(b)
		90	>123(b)
I-4	Bright-Cd plated	75	>101(b)
		90	(c)
I-7	Dull-Cd plated	75	>139(b)
		90	>100(b)
I-11	Dull-Cd plated	75	>132(b)
		90	>116(b)
<u>AISI E 8740 Steel, NTS = 273,000 psi</u>			
K-1	Cleaned commercially	90	10.7
K-2	Cleaned commercially	90	13.8
K-7	Cleaned in laboratory	90	>191(b)
K-3	Bright-Cd plated	75	28.8(d)
K-4	Bright-Cd plated	75	44.6(d)
K-5	Bright-Cd plated	75	67.7(d)
K-9	Dull-Cd plated	75	>119(b)
		90	>100(b)
K-11	Dull-Cd plated	75	>178(b)
		90	32.5
K-10	Dull-Cd plated	90	>113(b)

(a) NTS = Notched tensile strength.

(b) Runout time = 100 hours; specimen did not fail.

(c) Specimen failed during loading.

(d) Specimen failed at button end instead of at the notch.

TABLE 9. RESULTS OF HYDROGEN ANALYSES OF CLEANED AND CADMIUM-PLATED SPECIMENS

Sample Number	Material	Condition	Average Hydrogen Content, ppm ^(a)
A-1	AISI Type H-11 steel	Cleaned commercially	0.2
A-3	Ditto	Cleaned in laboratory	1.2
A-2	"	Bright-Cd plated	1.1
A-4	"	Dull-Cd plated	3.5
B-1	AISI 4130 steel	Cleaned commercially	0.2
B-3	Ditto	Cleaned in laboratory	0.7
B-2	"	Bright-Cd plated	0.6
B-4	"	Dull-Cd plated	3.7
C-1	AISI 4340 steel	Cleaned commercially	1.9 ^(b)
C-3	Ditto	Cleaned in laboratory	1.0
C-2	"	Bright-Cd plated	0.4 ^(b)
C-4	"	Dull-Cd plated	5.0
I-1	18Ni maraging steel	Cleaned commercially	1.6 ^(b)
I-3	Ditto	Cleaned in laboratory	1.1
I-2	"	Bright-Cd plated	0.6 ^(b)
I-4	"	Dull-Cd plated	2.8
K-1	AISI E 8740 steel	Cleaned commercially	0.2
K-3	Ditto	Cleaned in laboratory	1.0
K-2	"	Bright-Cd plated	1.0
K-4	"	Dull-Cd plated	3.1

(a) All values ± 0.1 ppm (precision of analysis).

(b) Values indicate a mixup in specimen identification.

TABLE 10. RESULTS OF HYDROGEN-STRESS-CRACKING EXPERIMENTS
EMPLOYING CLEANED AND WOOD'S-NICKEL-STRIKE-
PLATED STAINLESS STEEL SPECIMENS

Sample Number	Condition	Applied Stress, percent of NTS ^(a)	Time for Failure, hr
<u>17-7 PH Stainless Steel, NTS = 199,000 psi</u>			
D-2	Cleaned commercially	90	>100 ^(b)
D-1	Nickel strike plated	75	>100 ^(b)
		90	>112 ^(b)
D-4	Nickel strike plated	75	>100 ^(b)
		90	2.9
D-3	Nickel strike plated	90	(c)
<u>17-4 PH Stainless Steel, NTS = 306,000 psi</u>			
E-1	Cleaned commercially	90	>100 ^(b)
E-2	Nickel strike plated	75	>101 ^(b)
		90	>111 ^(b)
E-5	Nickel strike plated	75	>121 ^(b)
		90	>116 ^(b)
<u>AM-355 Stainless Steel, NTS = 268,000 psi</u>			
G-2	Cleaned commercially	90	>100 ^(b)
G-3	Nickel strike plated	75	>100 ^(b)
		90	>102 ^(b)
G-6	Nickel strike plated	75	>130 ^(b)
		90	>116 ^(b)
G-4	Nickel strike plated	90	>169 ^(b)

(a) NTS = Notched tensile strength.

(b) Runout time 100 hours; specimen did not fail.

(c) Specimen failed during loading.

The failure of the other materials at hydrogen contents below the critical amount determined in Phase 1 is attributed to the presence of the notch, which acted as a stress concentrator. Except for the AISI E 8740 steel, these other alloys failed only at applied stresses of 90 percent of their respective notched-tensile strengths. This loading condition is more severe than the loading condition used in the Phase 1 experiments and, consequently, the failure of the alloys at this higher applied stress with lower hydrogen contents is not unexpected.

The results of the hydrogen-stress-cracking experiments to evaluate the stainless steel specimens that had been cleaned and then electroplated in a Wood's-nickel-strike bath are listed in Table 10. The hydrogen analyses of specimens of these materials that had received the same processing are listed in Table 11.

TABLE 11. RESULTS OF HYDROGEN ANALYSES OF CLEANED AND NICKEL-STRIKE-PLATED STAINLESS STEEL SPECIMENS

Sample Number	Material	Condition	Average Hydrogen Content, ppm ^(a)
D-1	17-7 PH stainless steel	Cleaned commercially	1.8
D-2	17-7 PH stainless steel	Nickel strike plated	5.2
E-1	17-4 PH stainless steel	Cleaned commercially	1.6
E-2	17-4 PH stainless steel	Nickel strike plated	5.4
G-1	AM-355 stainless steel	Cleaned commercially	2.4
G-2	AM-355 stainless steel	Nickel strike plated	5.6

(a) All values ± 0.1 ppm by weight (precision of analysis).

The results of the hydrogen-stress-cracking experiments indicated that only the 17-7 PH steel was embrittled by the Wood's-nickel-strike plating process. This alloy was the least susceptible of these three alloys to hydrogen-stress cracking in the Phase 1 experiments. However, it was the most notch sensitive of these three alloys as determined by the notched tensile stress. Presumably, this notch sensitivity was partially responsible for the increased susceptibility to hydrogen-stress cracking.

The hydrogen analyses again showed that the plating process was responsible for introducing more hydrogen into the specimens than the preplating cleaning processes. Comparison of these hydrogen contents with those of specimens cathodically charged under the limiting conditions for hydrogen-stress cracking shows that only the 17-4 PH stainless steel contained more hydrogen after electroplating than the amount that produced failure under the cathodic charging conditions. Both of the other alloys contained somewhat less hydrogen after electroplating than after cathodic charging. The lack of failure exhibited by the 17-4 PH steel may be attributed to the fact that the nickel-strike electroplate was very thin and, consequently, allowed sufficient hydrogen to effuse from the specimen before the failure mechanism could become operative, so that fracture could not occur. Apparently, the hydrogen picked up by the AM-355 stainless steel during cleaning was not sufficient to initiate failure.

CONCLUSIONS

The results of this evaluation of the susceptibilities of various alloys to hydrogen-stress cracking have resulted in the following observations and conclusions:

(1) The Ti-6Al-4V, Alloy 718, Waspaloy, René 41, and U-212 steel were not susceptible to hydrogen-stress cracking at relatively high applied stresses under the most severe cathodic charging conditions used.

(2) All the other alloys were susceptible to hydrogen-stress cracking at relatively high applied stresses under the most severe cathodic charging condition used. As the severity of the charging condition was reduced, differences in the relative susceptibilities of the alloys to failure became more apparent. On the basis of time to failure under the various charging conditions, the alloys can be listed in the following order of increasing susceptibility to failure: 17-7 PH stainless steel, AISI E 8740 steel, 18Ni maraging steel, AM-355 stainless steel, 17-4 PH stainless steel, AISI Type H-11 tool steel, AISI 4130 steel, and AISI 4340 steel.

(3) There is no simple correlation between the rate at which a material accepts hydrogen and its susceptibility to failure. However, for six of the eight susceptible alloys for which the evaluation was completed, the susceptibility to hydrogen-stress cracking increased with increasing hydrogen-entry rate.

(4) Although the alloys contained different amounts of hydrogen under conditions that produced failure, there appeared to be little relationship between the average hydrogen content under limiting charging conditions for hydrogen-stress cracking and sensitivity to cracking. It would appear that hydrogen distribution within the specimen is a more important factor in hydrogen-stress cracking than is average hydrogen content.

(5) Conventional cleaning processes can introduce sufficient hydrogen to cause embrittlement in susceptible alloys.

(6) Conventional bright- and dull-cadmium plating processes can introduce sufficient hydrogen to cause embrittlement in susceptible alloys. The alloys evaluated by these processes can be listed in the following order of increasing degree of embrittlement: 18Ni maraging steel, AISI Type H-11 tool steel, AISI E 8740 steel, AISI 4130 steel, and AISI 4340 steel.

(7) The 17-7 PH steel was embrittled by electroplating in a Wood's-nickel-strike bath, while the 17-4 PH stainless steel and the AM-355 stainless steel were not embrittled by this treatment.

(8) The cadmium-electroplating processes and the Wood's-nickel-strike-electroplating process generally introduced more hydrogen into the specimens than did the preplating cleaning processes.

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